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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:	Maurice Clarence Kemp, Robert Blaine Lalum, Zhong Wei Xie, Michael Anthony Cunha, Robert H. Carpenter, Zhang Shu, Yao Yu, and David E. Lewis
Serial No.:	09/500,473
Filed:	February 9, 2000
For:	ACIDIC SOLUTION OF SPARINGLY-SOLUBLE GROUP IIA COMPLEXES
Group No.:	1761
Examiner:	Robert A. Madsen

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. § 1.132

ROBERT BLAINE LALUM declares that:

1. I am a co-inventor of, and familiar with, the present U.S. Patent Application Serial No. 09/500,473, filed Feb. 9, 2000, in the name of Maurice C. Kemp, et al., and entitled "Acidic Solution of Sparingly-Soluble Group IIA Complexes." I am also familiar with the Official Action dated May 6, 2004 issued therein.

2. I am over 21 years old. I have worked as a research scientist specializing in acidic chemical formulations for the last 6 years for Mionix Corporation, assignee of the present application. I graduated from California Polytechnic State University in San Luis Obispo in June 1998 with a BS in Animal Science and a heavy concentration in courses on pre-veterinary medicine, biology, and chemistry.

3. I personally prepared and conducted experiments described below to demonstrate the differences between the AGIIS of the present invention and the acid replacement solutions of U.S. Patent No. 5,830,838 to Wurzburger, et al. ("Wurzburger") and U.S. Patent No. 5,895,782 to Overton et al. ("Overton"). I reproduced, to the best I could, thirteen (13) different methods disclosed and/or claimed in Wurzburger and Overton. These methods for producing the acid replacement solutions disclosed and/or claimed in Wurzburger and Overton are described below as Experimental Procedures #1 – #13.

4. **Experimental Procedure #1** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 1 – 14. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 2 moles of sulfuric acid, which is equal to 196.16 g, or 206.48 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 1 L, or 1 kg, of deionized water was measured into a second flask;

(d) 1 mole of calcium hydroxide, which is equal to 74.09 g, or 75.60 g of 98% pure calcium hydroxide after making a purity compensation, was added to the second flask;

(e) 2 moles of potassium hydroxide, which is equal to 112.22 g, or 132.02 g of 85% pure potassium hydroxide after making a purity compensation, was added to the second flask;

(f) the sulfuric acid of the first flask was slowly added into the contents of the second flask at a rate of 100 mL every 10 minutes until all the contents were added;

(g) the solution was stirred until the reaction was complete; and

(h) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution itself contained a large amount of precipitate and had a tan and cloudy appearance.

5. **Experimental Procedure #2** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 1 – 14. The steps were carried out as follows:

(a) 2 L, or 2 kg, of deionized water was measured into a first flask;

(b) 2 moles of sulfuric acid, which is equal to 196.16 g, or 206.48 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 1 L, or 1 kg, of deionized water was measured into a second flask;

(d) 1 mole of calcium hydroxide, which is equal to 74.09 g, or 75.60 g of 98% pure calcium hydroxide after making a purity compensation, was added to the second flask;

(e) 2 moles of potassium hydroxide, which is equal to 112.22 g, or 132.02 g of 85% pure potassium hydroxide after making a purity compensation, was added to the second flask;

(f) the sulfuric acid of the first flask was slowly added into the contents of the second flask at a rate of 100 mL every 10 minutes until all the contents were added;

(g) the solution was stirred until the reaction was complete; and

(h) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution itself contained a large amount of precipitate and had a tan and cloudy appearance.

6. **Experimental Procedure #3** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 1 – 14. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 2 moles of sulfuric acid, which is equal to 196.16 g, or 206.48 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 2 L, or 2 kg, of deionized water was measured into a second flask;

(d) 1 mole of calcium hydroxide, which is equal to 74.09 g, or 75.60 g of 98% pure calcium hydroxide after making a purity compensation, was added to the second flask;

(e) 2 moles of potassium hydroxide, which is equal to 112.22 g, or 132.02 g of 85% pure potassium hydroxide after making a purity compensation, was added to the second flask;

(f) the sulfuric acid of the first flask was slowly added into the contents of the second flask at a rate of 100 mL every 10 minutes until all the contents were added;

(g) the solution was stirred until the reaction was complete; and

(h) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution itself contained a large amount of precipitate and had a tan and cloudy appearance.

7. **Experimental Procedure #4** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 1 – 4, and an embodiment disclosed in Wurzburger, at Col. 1, lines 57 – 67. The steps were carried out as follows:

(a) 2 L, or 2 kg, of deionized water was measured into a first flask;

(b) 2 moles of sulfuric acid, which is equal to 196.16 g, or 206.48 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 2 L, or 2 kg, of deionized water was measured into a second flask;

(d) 1 mole of calcium hydroxide, which is equal to 74.09 g, or 75.60 g of 98% pure calcium hydroxide after making a purity compensation, was added to the second flask;

(e) 2 moles of potassium hydroxide, which is equal to 112.22 g, or 132.02 g of 85% pure potassium hydroxide after making a purity compensation, was added to the second flask;

(f) the sulfuric acid of the first flask was slowly added into the contents of the second flask at a rate of 100 mL every 10 minutes until all the contents were added;

(g) the solution was stirred until the reaction was complete; and

(h) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution itself contained a large amount of precipitate and had a tan and cloudy appearance.

8. **Experimental Procedure #5** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 17 – 24. The steps were carried out as follows:

(a) 2 L, or 2 kg, of deionized water was measured into a first flask;

(b) 1 mole of sulfuric acid, which is equal to 98.08 g, or 103.2 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 1 mole of calcium metal, which is equal to 40.08 g, or 40.48 g of 99% pure calcium metal after making a purity compensation, was slowly added to the second flask in the following increments: 6.8 g, 5.5 g, 5.0 g, 5.8 g, 6.5 g, 5.3 g, and 5.5 g, for a total of 40.4 g; and

(d) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution contained a large amount of unreacted calcium metal which was

encapsulated by calcium salts. When the precipitate was isolated and finely ground, it demonstrated a pH of greater than 10.

9. **Experimental Procedure #6** This procedure corresponded to an embodiment disclosed in Overton, at Col. 2, lines 17 – 24. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 1 mole of sulfuric acid, which is equal to 98.08 g, or 103.2 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 1 mole of calcium metal, which is equal to 40.08 g, or 40.48 g of 99% pure calcium metal after making a purity compensation, was slowly added to the second flask in the following increments: 4.5 g, 4.9 g, 4.8 g, 5.2 g, 5.5 g, 5.1 g, 3.9 g, and 6.5 g, for a total of 40.4 g; and

(d) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution contained a large amount of unreacted calcium metal which was encapsulated by calcium salts. When the precipitate was isolated and finely ground, it demonstrated a pH of greater than 10.

10. **Experimental Procedure #7** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 0.5 L, or 500 g, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 0.5 moles of calcium hydroxide, which is equal to 37.05 g, or 37.80 g of 98% pure calcium hydroxide after making a purity compensation, was slowly added to the first flask;

(d) 1 mole of potassium hydroxide, which is equal to 56.11 g, or 66.01 g of 85% pure potassium hydroxide after making a purity compensation, was slowly added to the first flask, so that the addition of both the calcium hydroxide and the potassium hydroxide took about 20 minutes; and

(e) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. Because the claimed procedure did not call for pre-mixing the base in a quantity of water, there were noticeable large particles of non-reacted base.

11. **Experimental Procedure #8** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 0.5 moles of calcium hydroxide, which is equal to 37.05 g, or 37.80 g of 98% pure calcium hydroxide after making a purity compensation, was slowly added to the first flask;

(d) 1 mole of potassium hydroxide, which is equal to 56.11 g, or 66.01 g of 85% pure potassium hydroxide after making a purity compensation, was slowly added to the first flask, so that the addition of both the calcium hydroxide and the potassium hydroxide took about 20 minutes; and

(e) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. Because the claimed procedure did not call for pre-mixing the base in a quantity of water, there were noticeable large particles of non-reacted base.

12. **Experimental Procedure #9** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 0.5 L, or 500 g, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) about 0.5 moles of calcium metal, which is equal to 20.04 g, or 20.24 g of 99% pure calcium metal after making a purity compensation, was slowly added to the second flask over a period of about 20 minutes in the following increments: 5.0 g, 4.3 g, 5.8 g, and 5.3 g, for a total of 20.4 g; and

(d) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution contained a large amount of unreacted calcium metal which was encapsulated by calcium salts.

13. **Experimental Procedure #10** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 0.5 moles of calcium metal, which is equal to 20.04 g, or 20.24 g of 99% pure calcium metal after making a purity compensation, was slowly added to the second flask over a period of about 20 minutes in the following increments: 5.0 g, 4.5 g, 5.0 g, and 5.7 g, for a total of 20.2 g; and

(d) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution contained a large amount of unreacted calcium metal which was encapsulated by calcium salts.

14. **Experimental Procedure #11** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 0.5 L, or 500 g, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 0.5 moles of calcium oxide, which is equal to 28.04 g, or 28.61 g of 98% pure calcium hydroxide after making a purity compensation, was slowly added to the first flask;

(d) 1 mole of potassium hydroxide, which is equal to 56.11 g, or 66.01 g of 85% pure potassium hydroxide after making a purity compensation, was slowly added to the first flask, so that the addition of both the calcium oxide and the potassium hydroxide took about 20 minutes; and

(e) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution and precipitate were very white or cloudy in appearance, and there was a high level of hydrated crystals. The solution contained a large amount of unreacted calcium oxide.

15. **Experimental Procedure #12** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 1 L, or 1 kg, of deionized water was measured into a first flask;

(b) 0.5 moles of sulfuric acid, which is equal to 49.04 g, or 51.62 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 0.5 moles of calcium oxide, which is equal to 28.04 g, or 28.61 g of 98% pure calcium hydroxide after making a purity compensation, was slowly added to the first flask;

(d) 1 mole of potassium hydroxide, which is equal to 56.11 g, or 66.01 g of 85% pure potassium hydroxide after making a purity compensation, was slowly added to the first flask, so that the addition of both the calcium oxide and the potassium hydroxide took about 20 minutes; and

(e) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution had a slight tan or yellow tint, due to the high amount of dissolved potassium sulfate. The solution contained a large amount of unreacted calcium oxide.

16. **Experimental Procedure #13** This procedure corresponded to Claim 1 of Wurzburger. The steps were carried out as follows:

(a) 2 L, or 2 kg, of deionized water was measured into a first flask;

(b) 2 moles of sulfuric acid, which is equal to 196.16 g, or 206.48 g of 95% pure sulfuric acid after making a purity compensation, was added to the first flask;

(c) 2 L, or 2 kg, of deionized water was measured into a second flask;

(d) 1 mole of calcium oxide, which is equal to 56.08 g, or 57.22 g of 98% pure calcium hydroxide after making a purity compensation, was added to the second flask;

(e) 2 moles of potassium hydroxide, which is equal to 112.22 g, or 132.02 g of 85% pure potassium hydroxide after making a purity compensation, was added to the second flask;

(f) the sulfuric acid of the first flask was slowly added into the contents of the second flask at a rate of 100 mL every 10 minutes until all the contents were added;

(g) the solution was stirred until the reaction was complete; and

(h) the completed solution was filtered using a 10 micron filter.

250 mL of the filtered solution was retained for ICP and HPLC analysis, which appears in Table 1 below. The solution contained a considerable amount of unreacted calcium oxide.

17. For comparison, four sample solutions of AGIIS of the present invention were prepared, having normalities of about 1.5 N, 5 N, 27 N, and 29 N. (Top four rows in Table 1)

18. The solutions prepared according to Experimental Procedures 1 – 13, as well as the four sample solutions of AGIIS, were analyzed by Inductive Coupled Plasma ("ICP" using ICP Spectrometer JY Ultima 2 with Version 5 Software from JY Horiba Corporation) to determine calcium ion concentration and potassium ion concentration. The solutions were analyzed by High Pressure Liquid Chromatography ("HPLC," using DX500 Chromatography System (Dionex Corporation) with PeakNet Software Version 5.2) to determine sulfate ion concentration. pH and normality of each sample were also determined. Negative pH values were calculated from the normality using the equation $pH = - \log [H+]$. A pH value greater than 12.00 was calculated from the normality using the equation $14 = pH + pOH$. The units "ppm" represent parts per million (mg/L). The results appear in Table 1 below.

Table 1

Solution	pH	Normality	Sulfate (ppm)	Calcium (ppm)	Potassium (ppm)
AGIIS (1.5 N)	-0.176	1.50(acidic)	71911.55	718.636	0.485
AGIIS (5 N)	-0.709	5.12(acidic)	250168.28	317.153	1.248
AGIIS (27 N)	-1.44	27.63(acidic)	1335792.93	995.66	30.138
AGIIS (29 N)	-1.47	29.65(acidic)	1455057.05	1931.88	1.698
Procedure # 1	3.28	5.25 X 10-7 (acidic)	27072.54	182.664	27601.995
Procedure # 2	11.95	0.009 (basic)	24850.59	320.142	21527.44
Procedure # 3	11.78	0.006 (basic)	23672.07	335.478	24423.796
Procedure # 4	7.07	Neutral	23353.18	287.322	22544.484
Procedure # 5	1.03	0.093 (acidic)	8323.56	627.417	4.428
Procedure # 6	0.04	0.92 (acidic)	45093.50	725.259	2.931
Procedure # 7	13.28	0.192 (basic)	52067.07	66.647	46216.358
Procedure # 8	13.07	0.117 (basic)	36100.98	109.345	35157.958
Procedure # 9	-0.04	1.098 (acidic)	54063.44	755.774	49.042
Procedure # 10	0.25	0.56 (acidic)	28200.94	704.606	56.298
Procedure # 11	13.38	0.24 (basic)	50089.80	128.670	47990.696
Procedure # 12	12.72	0.053 (basic)	40926.74	94.264	32979.74
Procedure # 13	10.69	4.90 X 10-7 (basic)	24042.29	419.275	18489.144

19. The comparison of the sample solutions of AGIIS with the thirteen solutions prepared according to Wurzburger and Overton demonstrates that the solutions disclosed in Wurzburger and Overton are significantly different from those of the current patent application.

20. The analysis of the thirteen solutions prepared according to Wurzburger and Overton above demonstrates that the acid replacement solutions disclosed in these patents do not possess a pH less than two, as the current claims require. The only four solutions to obtain a pH of less than two, namely those produced by Procedures 5, 6, 9, and 10, appear to have done so because of the incomplete reaction between the calcium metal and the acid.

21. The comparison of the sample solutions of AGIIS with the thirteen solutions prepared according to Wurzburger and Overton demonstrates that the solutions disclosed in Wurzburger and Overton have significantly lower amounts of sulfate ions than the AGIIS of the current claims.

22. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Robert Blaine Lalum

11/14/04
Date